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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C11D 17/00, 3/00	A1	(11) International Publication Number: WO 99/64556 (43) International Publication Date: 16 December 1999 (16.12.99)
<p>(21) International Application Number: PCT/IB99/00996</p> <p>(22) International Filing Date: 1 June 1999 (01.06.99)</p> <p>(30) Priority Data: 60/088,170 5 June 1998 (05.06.98) US</p> <p>(71) Applicant (for all designated States except US): THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US).</p> <p>(72) Inventor; and (75) Inventor/Applicant (for US only): PARRY, Diane [US/US]; 9568 Brehm Road, Cincinnati, OH 45252 (US).</p> <p>(74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217-1087 (US).</p>		<p>(81) Designated States: BR, CA, CN, IN, JP, MX, US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).</p> <p>Published <i>With international search report.</i></p>
<p>(54) Title: NON-AQUEOUS, LIQUID DETERGENT COMPOSITIONS CONTAINING GASIFIED PARTICULATE MATTER</p> <p>(57) Abstract</p> <p>Non-aqueous, particulate-containing liquid laundry detergent compositions which are in the form of a stable suspension of particulate material. These compositions comprise gasified particles that are solid at room temperature, highly water soluble, and essentially insoluble in the non-aqueous liquid detergent compositions. Preferably these non-aqueous liquid detergent compositions additionally comprise a peroxygen bleaching agent and an organic detergent builder, dispersed in a liquid phase preferably structured with a surfactant. Such compositions provide especially desirable cleaning and bleaching of fabrics laundered therewith and also exhibit especially desirable aesthetics.</p>		

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NON-AQUEOUS, LIQUID DETERGENT COMPOSITIONS CONTAINING GASIFIED PARTICULATE MATTER

FIELD OF THE INVENTION

This invention relates to liquid laundry detergent products which are non-aqueous in nature and which are in the form of stable dispersions of particulate material which include gasified particles and which preferably also includes other materials such as bleaching agents and/or conventional detergent composition adjuvants.

BACKGROUND OF THE INVENTION

Non-aqueous liquid detergent products have many advantages over aqueous based liquid detergent products. But there are some drawbacks to compositions that comprise little or no water. For example, when non-aqueous detergents are poured into an aqueous wash solution, the non-aqueous detergent often does not disperse immediately. This results in poor or untimely dissolution of the active detergent ingredients. This problem is much less pronounced in aqueous based solutions, which mix readily with wash water. Nonetheless, numerous methods have been proposed to improve dispersion of aqueous liquid laundry detergents in water, but few of these are suitable for a non-aqueous system. Likewise, some of the methods used to improve the dispersion of granular detergent products are of limited value with respect to improving the dispersion of a non-aqueous liquid in an aqueous medium.

A second problem facing the commercial marketing of laundry detergent products is differentiating one given product from other commercially available products of the same general type. Colored speckles are sometimes used to create such distinctiveness. Generally, speckles in detergent products should be larger than 200 microns to be easily visible to the consumer. But in a liquid detergent context, and especially with respect to non-aqueous liquid detergents, it is a challenge to stably suspend particles that are larger than 400 microns. The formulator must use materials which are not soluble in the non-aqueous liquid product yet are soluble in the laundry wash water. Materials which meet this criteria for speckles are generally salts or polymers. But the density of most salts is significantly greater than that of the non-aqueous liquid detergents in which they are to be suspended. Thus, colored speckles made from salts tend to sediment or settle out of non-aqueous liquid detergent products, and many deaerated polymers exhibit the same problem.

In addition to a detergent product's aesthetic qualities, it is well known that consumers associate certain product "signals" with product performance. These signals can be audible, olfactory, tactile, or visual. For example, the amount of foam or suds produced by a detergent

when it is added to the wash water is often associated with the product's cleaning performance; even if there is no direct correlation between the amount of foam or suds produced and how well the fabrics articles are cleaned. Thus, the "signals" delivered to the consumer are an important consideration when formulating any detergent composition.

Moreover, it is often desirable to add perfumes to liquid laundry detergent products in an effort to scent the fabrics that are laundered therewith. Unfortunately, when the amount of perfume required to scent a wash load of fabrics is in the concentrated liquid laundry detergent, the detergent can have a very strong smell. Encapsulation of the perfume has been used in the past to combat this problem, but this requires the manufacture and addition of a separate particle. And for the encapsulated particle to release the perfume in an aqueous wash solution, the encapsulation material must be water soluble; making the encapsulated particle unsuitable for an aqueous based liquid laundry detergent.

Thus, the problems of product dispersion to increase the rate of solubility, product differentiation, product signaling, and adding sufficient perfume are persistent problems for formulators of non-aqueous liquid detergent products. Accordingly, it is an object of the present invention to formulate non-aqueous heavy-duty detergent compositions having improved dispersion and solubility properties.

It is a further object of this invention to improve the dispersion properties of non-aqueous detergents with a gasified particle that improves the product aesthetics, that can be suspended in the non-aqueous solution, that will provide a favorable signal to the consumer, and that can also be used to introduce perfumes.

It is a further object of the present invention to stably suspend the gasified particles in non-aqueous liquid detergent products.

It is a further object of the present invention to provide gasified particles that impart desirable aesthetics to concentrated liquid detergent products but do not interfere with the laundering operations that use such products.

Surprisingly it has been found that there is a small class of materials that have the requisite properties to serve as gasified particles and that can be used to achieve the forgoing objectives with respect to the non-aqueous liquid detergent products of this invention.

SUMMARY OF THE INVENTION

The present invention provides non-aqueous liquid detergent compositions comprising a stable suspension of solid, substantially insoluble particulate material dispersed throughout a non-aqueous, surfactant-containing liquid phase. Such compositions comprise: A) from about 49% to 99.95% by weight of the composition of a surfactant-containing, preferably structured, non-aqueous liquid phase; and B) from about 0.05% to about 5%, preferably from about 0.1% to

about 3%, and most preferably from about 0.2% to about 2%, by weight of the composition of gasified particles that are substantially insoluble in the non-aqueous liquid phase; solid at about 25°C ("room temperature"), preferably, solid up to about 40°C, and highly water soluble.

Preferably these compositions further comprise from about 1% to 50% by weight of the composition of additional insoluble particulate material that ranges in size from about 0.1 to 1500 microns, that is substantially insoluble in said liquid phase and that is selected from peroxygen bleaching agents, bleach activators, organic detergent builders and inorganic alkalinity sources, colored speckles and combinations thereof. Even more preferably the surfactant-containing non-aqueous liquid phase has a density of from about 0.6 to 1.4 g/cc.

In one preferred embodiment of the present invention the surfactant-containing liquid phase is formed by combining: i) from about 1% to about 80% by weight of the liquid phase of one or more non-aqueous organic diluents; and ii) from about 20% to 99% by weight of the liquid phase of a surfactant selected from anionic, nonionic and cationic surfactants and combinations thereof.

Non-aqueous liquid detergent products made according to this invention have the surprising benefit of superior product dispersion that increases the rate of product dissolution in the wash water. Additionally, the gasified particles can be used to add desirable product aesthetics in the form of colored speckles.

Yet another advantage of the present invention is that the gasified particles can provide both audible and olfactory signals to the consumer that the product is working. Specifically, as is discussed in greater detail below, the gasified particles of this invention give off a "popping" and/or a "hissing" sound as they dissolve. These sounds let the consumer know that the product is dissolving and, thus, beginning to work. And when the pressurized gas comprises a perfume, the perfume is emitted as the particles dissolve, giving the consumer an olfactory signal that the product is beginning to work.

DETAILED DESCRIPTION OF THE INVENTION

The non-aqueous liquid detergent compositions of this invention comprise a surfactant-containing, preferably surfactant-structured liquid phase in which solid, substantially insoluble gasified particles are suspended. The components of the liquid phase, the gasified particles and optional materials of the detergent compositions herein, as well as composition form, preparation and use, are described in greater detail as follows: (All concentrations and ratios are on a weight basis unless otherwise specified.)

GASIFIED PARTICLES

The compositions of this invention comprise from about 0.05% to about 5%, preferably from about 0.1% to about 3%, and most preferably from about 0.2% to about 2%, by weight of the composition of gasified particles that are substantially insoluble in the non-aqueous liquid phase; solid at about 25°C ("room temperature"), and highly water soluble. The gasified particles comprise a core material that encapsulates a pressurized gas, optionally, the particles can be dyed, coated and perfumes can be added. It is understood that all of the materials used in the gasified particles should be compatible with a fabric laundering process because the particles will eventually dissolve, releasing the ingredients into the wash water. For example, certain dyes may preferentially deposit on fabrics rather than be rinsed out in the wash water. Dyes of this type would be less preferred than dyes that readily dissolve in the wash water and are rinsed away.

As used herein, the term "gasified particles" is intended to exclude particles that evolve gas as a result of a chemical reaction, for example, the reaction of sodium bicarbonate with water resulting in the evolution of carbon dioxide. The particles of this invention comprise a pressurized gas that escapes as the core material dissolves or is shattered.

The pressurized gas trapped within the gasified particles of this invention is preferably selected from the group consisting of carbon dioxide, nitrogen, oxygen, helium, hydrogen, air, argon, neon, chlorine and mixtures thereof. The core material of these gasified particles is preferably a glassy solid and most preferably comprises materials selected from the group consisting of sucrose, lactose, glucose, fructose, galactose, maltose, polyethylene glycol, polyvinyl alcohol, fatty acids, and mixtures thereof. It is understood that certain fatty acids will not be solid at room temperature. Therefore, if used to make gasified particles of this invention, fatty acids should have a high melting point or should be mixed with other materials to raise the melting point. The most preferred core materials for the gasified particles of this invention are sucrose and polyethylene glycol that has a molecular weight between about 2,000 and 20,000.

The gasified particles can additionally comprise a dye or pigment, preferably selected from the group consisting of ultramarine blue, indigo carmine, FD&C blue 1, D&C yellow 5, D&C yellow 6, D&C red 21, D&C red 27, D&C orange 5, bromo acid dyes, sodium fluorescein, liquitint bright blue, liquitint bright yellow, duasyn blue and mixtures thereof.

It may be desirable to coat the gasified particles of this invention to, for example, modify the release time of the gas or to protect the particles from the non-aqueous liquid detergent composition. Any additional coatings on the gasified particles must be water soluble or dispersable, and the coating preferably comprises materials selected from the group

consisting of sucrose, lactose, glucose, fructose, galactose, maltose, polyethylene glycol, polyvinyl alcohol, fatty acids, and mixtures thereof.

The gasified particles should range in particle size between about 0.1 and about 1,500 microns, preferably between about 1 and about 1,000 microns, and most preferably between about 10 and about 400 microns. Moreover, the gasified particles preferably range in density between about 0.6 and 1.4 g/cc, more preferably between about 0.8 and 1.3 g/cc, and most preferably between about 1.0 and 1.3 g/cc.

The gasified particles of this invention can be manufactured by a variety of processes. In fact, processes for making gasified particles suitable for use herein are known to the art of candy making. Specifically, in various U.S. Patents, Nos. 3,985,909, 3,985,910, 4,001,457, and 4,289,794, all of which are assigned to the General Foods Corporation and are collectively referred to herein as "the General Foods patents", processes for making gasified candy are taught. The entire disclosure of each of the four General Foods Patents are hereby incorporated herein by reference. Necessarily, the processes taught in these gasified candy patents involve only edible, and typically flavored particulate material. But the gasified particles of this invention are not so limited. Moreover, certain dyes disclosed in the General Foods patents may be unsuitable for laundry applications. Having expressed these limitations, the processes taught in the General Foods patents are generally applicable to the production gasified particles of this invention.

In general, to produce the gasified particles of this invention the core material must be selected and then melted, that is, heated until it is in a molten state. Preferably, a minor amount of a liquid, for example, water or corn syrup, can be added to the molten material to achieve the desired consistency. A pressure vessel having polished inner walls is preferably used to melt the core material. Alternatively, the core material can be heated in any appropriate vessel and then transferred to a pressure vessel. The pressure vessel, in any event, must be configured to provide agitation, preferably in the form of a mixing blade attached to a shaft. Once in the pressure vessel, the gas is added and mixed into the molten core material. The gas can be added through one or more vents in the pressure vessel, or, more preferably, through ports in the mixer shaft. The pressure in the vessel is set to the pressure desired in the solidified gasified particles. Preferably, the gas pressure within the gasified particles is from about 50 psig to about 1,000 psig, more preferably from about 300 psig to about 1,000 psig, and most preferably from about 600 psig to about 1,000 psig. At the preferred pressure ranges the solid gasified particles comprise between about 0.5 and 15 milliliters of pressurized gas per gram of core material.

Perfumes can be added to the gasified particles by mixing a perfume ingredient with at least one of the molten core material and the pressurized gas. Preferably, the perfume is highly volatile so that it can be introduced in the pressurized gas. When the gasified particles begin to

dissolve, the pressurized gas escapes rapidly carrying the entrapped perfume with it. This results in a quick and pronounced "bloom" of perfume being emitted from the wash water shortly after the detergent composition is added. Consumers of detergent products have founds this immediate olfactory signal pleasing and reassuring.

Although virtually any perfume that is a liquid or gas at room temperature will work in the gasified particles of this invention, a listing of perfume ingredients of interest can be found in U.S. Patent No. 4,515,705, which issued on May 7, 1985, to Moeddel, and is assigned to The Procter and Gamble Company. The entire disclosure of the Moeddel patent is hereby incorporated herein by reference. It is understood that any perfume used must be compatible with the core material and the pressurized gas. For example, a perfume that dissolves the core material would generally be inappropriate for use in the present invention.

After the pressurized gas has been sufficiently mixed into the molten core material the molten mixture must be cooled until it solidifies. The cooling step can occur in the same pressure vessel that was used to form the mixture, or the molten mixture can be transferred to a different vessel. But if a separate cooling vessel is used to cool the molten mixture, the pressure in both vessels should be substantially the same to avoid premature migration of the gas from the molten core material. The criticality of the pressure in the cooling vessel will necessarily depend on the speed of cooling. If the molten mixture cools rapidly, the pressure in the cooling vessel will be less important. If a separate cooling vessel is used it should preferably have polished walls to facilitate complete removal of the solidified product.

When the mixture has solidified it can be removed from the cooling vessel by any appropriate means. One such method is to break the solid material into small pieces that can be easily removed from the cooling vessel. Regardless of how the solid material is removed from the cooling vessel the solid material should be broken-up into particles of the desired size. The particles can be formed by any appropriate means, for example, milling. After particles of the appropriate size are formed, they can be coated as discussed above, or they can be added directly to the non-aqueous detergent compositions of this invention.

In an alternate method, the core material can be melted and mixed with the pressurized gas in an extruder, or a mixture of molten core material and pressurized gas can be fed into an extruder. The molten material can be cooled before, during and/or after extrusion. Those skilled in the art of particle formulation will be able to select an extruder and process conditions without undue experimentation.

When the gasified particle containing non-aqueous detergents of this invention are added to an aqueous wash water solution, the core material of the gasified particles begins to dissolve. While not wanting to be bound by any one theory, it is believed that as the core material dissolves, the material enclosing the pressurized gas begins to thin and at some point

becomes too thin to entrap the pressurized gas. At this point the pressurized gas escapes by breaking through the walls of the particle, resulting in a small explosion. A "popping" sound is created as the particle walls are shattered. Additionally, some particles tend to "fizz" as gas escapes through small holes in the particle walls before, or while the particle walls explode. As the particles explode and the pressurized gas is released, the shattered pieces of the particles and the escaping gas both serve to break-up the surrounding non-aqueous detergent product. This results in faster dissolution of the non-aqueous detergent in the wash water.

Moreover, the size of the entrapped bubbles of gas will affect the sound given off by the gasified particles of this invention as they dissolve in the aqueous wash water. When using the materials and processes of this invention, the entrapped bubbles of gas will typically range in size from about 5 to about 300 microns. It has been observed that the core material often effects the size of the entrapped bubbles. For example, if all other process conditions are held constant, the bubbles trapped in polyethylene glycol are generally smaller than those trapped in a sucrose matrix. Fizzing typically result from entrapped bubbles whose size is in the range of from about 5 to about 100 microns, while popping generally occurs when the bubbles are greater than about 100 microns. Both the fizzing and popping of the gasified particles of this invention provide audible, and often visual signals to the consumer.

SURFACTANT-CONTAINING LIQUID PHASE

The surfactant-containing, non-aqueous liquid phase will generally comprise from about 49% to 99.95% by weight of the detergent compositions herein. More preferably, this liquid phase is surfactant-structured and will comprise from about 52% to 98.9% by weight of the compositions. Most preferably, this non-aqueous liquid phase will comprise from about 55% to 70% by weight of the compositions herein. Such a surfactant-containing liquid phase will frequently have a density of from about 0.6 to 1.4 g/cc, more preferably from about 0.9 to 1.3 g/cc. The liquid phase of the detergent compositions herein is preferably formed from one or more non-aqueous organic diluents into which is mixed a surfactant structuring agent which is preferably a specific type of anionic surfactant-containing powder.

(a) Non-aqueous Organic Diluents

The major component of the liquid phase of the detergent compositions herein comprises one or more non-aqueous organic diluents. The non-aqueous organic diluents used in this invention may be either surface active, i.e., surfactant, liquids or non-aqueous, non-surfactant liquids referred to herein as non-aqueous solvents. The term "solvent" is used herein to connote the non-surfactant, non-aqueous liquid portion of the compositions herein. While some of the essential and/or optional components of the compositions herein may actually dissolve in the

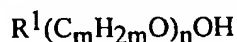
"solvent"-containing liquid phase, other components will be present as particulate material dispersed within the "solvent"-containing liquid phase. Thus the term "solvent" is not meant to require that the solvent material be capable of actually dissolving all of the detergent composition components added thereto.

The non-aqueous liquid diluent component will generally comprise from about 50% to 100%, more preferably from about 50% to 80%, most preferably from about 55% to 75%, of a structured, surfactant-containing liquid phase. Preferably the liquid phase of the compositions herein, i.e., the non-aqueous liquid diluent component, will comprise both non-aqueous liquid surfactants and non-surfactant non-aqueous solvents.

i) Non-aqueous Surfactant Liquids

Suitable types of non-aqueous surfactant liquids which can be used to form the liquid phase of the compositions herein include the alkoxylated alcohols, ethylene oxide (EO)-propylene oxide (PO) block polymers, polyhydroxy fatty acid amides, alkylpolysaccharides, and the like. Such normally liquid surfactants are those having an HLB ranging from 10 to 16. Most preferred of the surfactant liquids are the alcohol alkoxylate nonionic surfactants.

Alcohol alkoxylates are materials which correspond to the general formula:



wherein R^1 is a $C_8 - C_{16}$ alkyl group, m is from 2 to 4, and n ranges from about 2 to 12. Preferably R^1 is an alkyl group, which may be primary or secondary, that contains from about 9 to 15 carbon atoms, more preferably from about 10 to 14 carbon atoms. Preferably also the alkoxylated fatty alcohols will be ethoxylated materials that contain from about 2 to 12 ethylene oxide moieties per molecule, more preferably from about 3 to 10 ethylene oxide moieties per molecule.

The alkoxylated fatty alcohol materials useful in the liquid phase will frequently have a hydrophilic-lipophilic balance (HLB) which ranges from about 3 to 17. More preferably, the HLB of this material will range from about 6 to 15, most preferably from about 8 to 15.

Examples of fatty alcohol alkoxylates useful in or as the non-aqueous liquid phase of the compositions herein will include those which are made from alcohols of 12 to 15 carbon atoms and which contain about 7 moles of ethylene oxide. Such materials have been commercially marketed under the trade names Neodol 25-7 and Neodol 23-6.5 by Shell Chemical Company. Other useful Neodols include Neodol 1-5, an ethoxylated fatty alcohol averaging 11 carbon atoms in its alkyl chain with about 5 moles of ethylene oxide; Neodol 23-9, an ethoxylated primary $C_{12} - C_{13}$ alcohol having about 9 moles of ethylene oxide and Neodol 91-10, an ethoxylated $C_9 - C_{11}$ primary alcohol having about 10 moles of ethylene oxide. Alcohol ethoxylates of this type have also been marketed by Shell Chemical Company under the

Dobanol tradename. Dobanol 91-5 is an ethoxylated C₉-C₁₁ fatty alcohol with an average of 5 moles ethylene oxide and Dobanol 25-7 is an ethoxylated C₁₂-C₁₅ fatty alcohol with an average of 7 moles of ethylene oxide per mole of fatty alcohol.

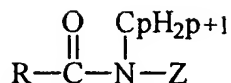
Other examples of suitable ethoxylated alcohols include Tergitol 15-S-7 and Tergitol 15-S-9 both of which are linear secondary alcohol ethoxylates that have been commercially marketed by Union Carbide Corporation. The former is a mixed ethoxylation product of C₁₁ to C₁₅ linear secondary alkanol with 7 moles of ethylene oxide and the latter is a similar product but with 9 moles of ethylene oxide being reacted.

Other types of alcohol ethoxylates useful in the present compositions are higher molecular weight nonionics, such as Neodol 45-11, which are similar ethylene oxide condensation products of higher fatty alcohols, with the higher fatty alcohol being of 14-15 carbon atoms and the number of ethylene oxide groups per mole being about 11. Such products have also been commercially marketed by Shell Chemical Company.

If alcohol alkoxylate nonionic surfactant is utilized as part of the non-aqueous liquid phase in the detergent compositions herein, it will preferably be present to the extent of from about 1% to 60% of the composition structured liquid phase. More preferably, the alcohol alkoxylate component will comprise about 5% to 40% of the structured liquid phase. Most preferably, an alcohol alkoxylate component will comprise from about 5% to 35% of the detergent composition structured liquid phase. Utilization of alcohol alkoxylate in these concentrations in the liquid phase corresponds to an alcohol alkoxylate concentration in the total composition of from about 1% to 60% by weight, more preferably from about 2% to 40% by weight, and most preferably from about 5% to 25% by weight, of the composition.

Another type of non-aqueous surfactant liquid which may be utilized in this invention are the ethylene oxide (EO) - propylene oxide (PO) block polymers. Materials of this type are well known nonionic surfactants which have been marketed under the tradename Pluronic. These materials are formed by adding blocks of ethylene oxide moieties to the ends of polypropylene glycol chains to adjust the surface active properties of the resulting block polymers. EO-PO block polymer nonionics of this type are described in greater detail in Davidsohn and Milwidsky; Synthetic Detergents, 7th Ed.; Longman Scientific and Technical (1987) at pp. 34-36 and pp. 189-191 and in U.S. Patents 2,674,619 and 2,677,700. All of these publications are incorporated herein by reference. These Pluronic type nonionic surfactants are also believed to function as effective suspending agents for the particulate material which is dispersed in the liquid phase of the detergent compositions herein.

Another possible type of non-aqueous surfactant liquid useful in the compositions herein comprises polyhydroxy fatty acid amide surfactants. Materials of this type of nonionic surfactant are those which conform to the formula:



wherein R is a C₉₋₁₇ alkyl or alkenyl, p is from 1 to 6, and Z is glyceryl derived from a reduced sugar or alkoxyated derivative thereof. Such materials include the C₁₂-C₁₈ N-methyl glucamides. Examples are N-methyl N-1-deoxyglucityl cocoamide and N-methyl N-1-deoxyglucityl oleamide. Processes for making polyhydroxy fatty acid, amides are known and can be found, for example, in Wilson, U.S. Patent 2,965,576 and Schwartz, U.S. Patent 2,703,798, the disclosures of which are incorporated herein by reference. The materials themselves and their preparation are also described in greater detail in Honsa, U.S. Patent 5,174,937, Issued December 26, 1992, which patent is also incorporated herein by reference.

The amount of total liquid surfactant in the preferred surfactant-structured, non-aqueous liquid phase herein will be determined by the type and amounts of other composition components and by the desired composition properties. Generally, the liquid surfactant can comprise from about 35% to 70% of the non-aqueous liquid phase of the compositions herein. More preferably, the liquid surfactant will comprise from about 50% to 65% of a non-aqueous structured liquid phase. This corresponds to a non-aqueous liquid surfactant concentration in the total composition of from about 15% to 70% by weight, more preferably from about 20% to 50% by weight, of the composition.

ii) Non-surfactant Non-aqueous Organic Solvents

The liquid phase of the detergent compositions herein may also comprise one or more non-surfactant, non-aqueous organic solvents. Such non-surfactant non-aqueous liquids are preferably those of low polarity. For purposes of this invention, "low-polarity" liquids are those which have little, if any, tendency to dissolve one of the preferred types of particulate material used in the compositions herein, i.e., the peroxygen bleaching agents, sodium perborate or sodium percarbonate. Thus relatively polar solvents such as ethanol are preferably not utilized. Suitable types of low-polarity solvents useful in the non-aqueous liquid detergent compositions herein do include non-vicinal C₄-C₈ alkylene glycols, alkylene glycol mono lower alkyl ethers, lower molecular weight polyethylene glycols, lower molecular weight methyl esters and amides, and the like.

A preferred type of non-aqueous, low-polarity solvent for use in the compositions herein comprises the non-vicinal C₄-C₈ branched or straight chain alkylene glycols. Materials of this type include hexylene glycol (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene glycol and 1,4-butylene glycol. Hexylene glycol is the most preferred.

Another preferred type of non-aqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C₂-C₃ alkylene glycol mono C₂-C₆ alkyl ethers. The specific

examples of such compounds include diethylene glycol monobutyl ether, tetraethylene glycol monobutyl ether, dipropylene glycol monoethyl ether, and dipropylene glycol monobutyl ether. Diethylene glycol monobutyl ether, dipropylene glycol monobutyl ether and butoxy-propoxy-propanol (BPP) are especially preferred. Compounds of the type have been commercially marketed under the tradenames Dowanol, Carbitol, and Cellosolve.

Another preferred type of non-aqueous, low-polarity organic solvent useful herein comprises the lower molecular weight polyethylene glycols (PEGs). Such materials are those having molecular weights of at least about 150. PEGs of molecular weight ranging from about 200 to 600 are most preferred.

Yet another preferred type of non-polar, non-aqueous solvent comprises lower molecular weight methyl esters. Such materials are those of the general formula: $R^1-C(O)-OCH_3$ wherein R^1 ranges from 1 to about 18. Examples of suitable lower molecular weight methyl esters include methyl acetate, methyl propionate, methyl octanoate, and methyl dodecanoate.

The non-aqueous, generally low-polarity, non-surfactant organic solvent(s) employed should, of course, be compatible and non-reactive with other composition components, e.g., bleach and/or activators, used in the liquid detergent compositions herein. Such a solvent component is preferably utilized in an amount of from about 1% to 70% by weight of the liquid phase. More preferably, a non-aqueous, low-polarity, non-surfactant solvent will comprise from about 10% to 60% by weight of a structured liquid phase, most preferably from about 20% to 50% by weight, of a structured liquid phase of the composition. Utilization of non-surfactant solvent in these concentrations in the liquid phase corresponds to a non-surfactant solvent concentration in the total composition of from about 1% to 50% by weight, more preferably from about 5% to 40% by weight, and most preferably from about 10% to 30% by weight, of the composition.

iii) Blends of Surfactant and Non-surfactant Solvents

In systems which employ both non-aqueous surfactant liquids and non-aqueous non-surfactant solvents, the ratio of surfactant to non-surfactant liquids, e.g., the ratio of alcohol alkoxylate to low polarity solvent, within a structured, surfactant-containing liquid phase can be used to vary the rheological properties of the detergent compositions eventually formed. Generally, the weight ratio of surfactant liquid to non-surfactant organic solvent will range about 50:1 to 1:50. More preferably, this ratio will range from about 3:1 to 1:3, most preferably from about 2:1 to 1:2.

(b) Surfactant Structurant

The non-aqueous liquid phase of the detergent compositions of this invention is prepared by combining with the non-aqueous organic liquid diluents hereinbefore described a surfactant which is generally, but not necessarily, selected to add structure to the non-aqueous liquid phase of the detergent compositions herein. Structuring surfactants can be of the anionic, nonionic, cationic, and/or amphoteric types.

Preferred structuring surfactants are the anionic surfactants such as the alkyl sulfates, the alkyl polyalkylate sulfates and the linear alkyl benzene sulfonates. Another common type of anionic surfactant material which may be optionally added to the detergent compositions herein as structurant comprises carboxylate-type anionics. Carboxylate-type anionics include the C₁₀-C₁₈ alkyl alkoxy carboxylates (especially the EO 1 to 5 ethoxycarboxylates) and the C₁₀-C₁₈ sarcosinates, especially oleoyl sarcosinate. Yet another common type of anionic surfactant material which may be employed as a structurant comprises other-sulfonated anionic surfactants such as the C₈-C₁₈ paraffin sulfonates and the C₈-C₁₈ olefin sulfonates. Structuring anionic surfactants will generally comprise from about 1% to 30% by weight of the compositions herein.

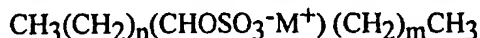
As indicated, one preferred type of structuring anionic surfactant comprises primary or secondary alkyl sulfate anionic surfactants. Such surfactants are those produced by the sulfation of higher C₈-C₂₀ fatty alcohols.

Conventional primary alkyl sulfate surfactants have the general formula



wherein R is typically a linear C₈ - C₂₀ hydrocarbyl group, which may be straight chain or branched chain, and M is a water-solubilizing cation. Preferably R is a C₁₀-C₁₄ alkyl, and M is alkali metal. Most preferably R is about C₁₂ and M is sodium.

Conventional secondary alkyl sulfates may also be utilized as a structuring anionic surfactant for the liquid phase of the compositions herein. Conventional secondary alkyl sulfate surfactants are those materials which have the sulfate moiety distributed randomly along the hydrocarbyl "backbone" of the molecule. Such materials may be depicted by the structure:

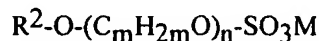


wherein m and n are integers of 2 or greater and the sum of m + n is typically about 9 to 15, and M is a water-solubilizing cation.

If utilized, alkyl sulfates will generally comprise from about 1% to 30% by weight of the composition, more preferably from about 5% to 25% by weight of the composition. Non-aqueous liquid detergent compositions containing alkyl sulfates, peroxygen bleaching agents, and bleach activators are described in greater detail in Kong-Chan et al.; WO 96/10073; Published April 4, 1996, which application is incorporated herein by reference.

Another preferred type of anionic surfactant material which may be optionally added to the non-aqueous cleaning compositions herein as a structurant comprises the alkyl

polyalkoxylate sulfates. Alkyl polyalkoxylate sulfates are also known as alkoxylated alkyl sulfates or alkyl ether sulfates. Such materials are those which correspond to the formula



wherein R^2 is a C_{10} - C_{22} alkyl group, m is from 2 to 4, n is from about 1 to 15, and M is a salt-forming cation. Preferably, R^2 is a C_{12} - C_{18} alkyl, m is 2, n is from about 1 to 10, and M is sodium, potassium, ammonium, alkylammonium or alkanolammonium. Most preferably, R^2 is a C_{12} - C_{16} , m is 2, n is from about 1 to 6, and M is sodium. Ammonium, alkylammonium and alkanolammonium counterions are preferably avoided when used in the compositions herein because of incompatibility with peroxygen bleaching agents.

If utilized, alkyl polyalkoxylate sulfates can also generally comprise from about 1% to 30% by weight of the composition, more preferably from about 5% to 25% by weight of the composition. Non-aqueous liquid detergent compositions containing alkyl polyalkoxylate sulfates, in combination with polyhydroxy fatty acid amides, are described in greater detail in Boutique et al; PCT Application No. PCT/US96/04223, which application is incorporated herein by reference.

The most preferred type of anionic surfactant for use as a structurant in the compositions herein comprises the linear alkyl benzene sulfonate (LAS) surfactants. In particular, such LAS surfactants can be formulated into a specific type of anionic surfactant-containing powder which is especially useful for incorporation into the non-aqueous liquid detergent compositions of the present invention. Such a powder comprises two distinct phases. One of these phases is insoluble in the non-aqueous organic liquid diluents used in the compositions herein; the other phase is soluble in the non-aqueous organic liquids. It is the insoluble phase of this preferred anionic surfactant-containing powder which can be dispersed in the non-aqueous liquid phase of the preferred compositions herein and which forms a network of aggregated small particles that allows the final product to stably suspend other additional solid particulate materials in the composition.

Such a preferred anionic surfactant-containing powder is formed by co-drying an aqueous slurry which essentially contains a) one of more alkali metal salts of C_{10-16} linear alkyl benzene sulfonic acids; and b) one or more non-surfactant diluent salts. Such a slurry is dried to a solid material, generally in powder form, which comprises both the soluble and insoluble phases.

The linear alkyl benzene sulfonate (LAS) materials used to form the preferred anionic surfactant-containing powder are well known materials. Such surfactants and their preparation are described for example in U.S. Patents 2,220,099 and 2,477,383, incorporated herein by reference. Especially preferred are the sodium and potassium linear straight chain alkylbenzene

sulfonates in which the average number of carbon atoms in the alkyl group is from about 11 to 14. Sodium C₁₁₋₁₄, e.g., C₁₂, LAS is especially preferred. The alkyl benzene surfactant anionic surfactants are generally used in the powder-forming slurry in an amount from about 20 to 70% by weight of the slurry, more preferably from about 20% to 60% by weight of the slurry.

The powder-forming slurry also contains a non-surfactant, organic or inorganic salt component that is co-dried with the LAS to form the two-phase anionic surfactant-containing powder. Such salts can be any of the known sodium, potassium or magnesium halides, sulfates, citrates, carbonates, sulfates, borates, succinates, sulfo-succinates and the like. Sodium sulfate, which is generally a bi-product of LAS production, is the preferred non-surfactant diluent salt for use herein. Salts which function as hydrotropes such as sodium sulfo-succinate may also usefully be included. The non-surfactant salts are generally used in the aqueous slurry, along with the LAS, in amounts ranging from about 1 to 50% by weight of the slurry, more preferably from about 5% to 40% by weight of the slurry. Salts that act as hydrotropes can preferably comprise up to about 3% by weight of the slurry.

The aqueous slurry containing the LAS and diluent salt components hereinbefore described can be dried to form the anionic surfactant-containing powder preferably added to the non-aqueous diluents in order to prepare a structured liquid phase within the compositions herein. Any conventional drying technique, e.g., spray drying, drum drying, etc., or combination of drying techniques, may be employed. Drying should take place until the residual water content of the solid material which forms is within the range of from about 0.5% to 4% by weight, more preferably from about 1% to 3% by weight.

The anionic surfactant-containing powder produced by the drying operation constitutes two distinct phases, one of which is soluble in the inorganic liquid diluents used herein and one of which is insoluble in the diluents. The insoluble phase in the anionic surfactant-containing powder generally comprises from about 10% to 45% by weight of the powder, more preferably from about 15% to 35% by weight of a powder.

The anionic surfactant-containing powder that results after drying can comprise from about 45% to 94%, more preferably from about 60% to 94%, by weight of the powder of alkyl benzene sulfonic acid salts. Such concentrations are generally sufficient to provide from about 0.5% to 60%, more preferably from about 15% to 60%, by weight of the total detergent composition that is eventually prepared, of the alkyl benzene sulfonic acid salts. The anionic surfactant-containing powder itself can comprise from about 0.45% to 45% by weight of the total composition that is eventually prepared. After drying, the anionic surfactant-containing powder will also generally contain from about 2% to 50%, more preferably from about 2% to 25% by weight of the powder of the non-surfactant salts.

After it is dried to the requisite extent, the combined LAS/salt material can be converted to flakes or powder form by any known suitable milling or comminution process. Generally at the time such material is combined with the non-aqueous organic solvents to form the structured liquid phase of the compositions herein, the particle size of this powder will range from 0.1 to 2000 microns, more preferably from about 0.1 to 1000 microns.

A structured, surfactant-containing liquid phase of the preferred detergent compositions herein can be prepared by combining the non-aqueous organic diluents hereinbefore described with the anionic surfactant-containing powder as hereinbefore described. Such combination results in the formation of a structured surfactant-containing liquid phase. Conditions for making this combination of preferred structured liquid phase components are described more fully hereinafter in the "Composition Preparation and Use" section. As previously noted, the formation of a structured, surfactant-containing liquid phase permits the stable suspension of colored speckles and additional functional particulate solid materials within the preferred detergent compositions of this invention.

ADDITIONAL SOLID PARTICULATE MATERIALS

In addition to the surfactant-containing liquid phase and the colored speckles, the non-aqueous detergent compositions herein also preferably comprise from about 1% to 50% by weight, more preferably from about 29% to 44% by weight, of additional solid phase particulate material which is dispersed and suspended within the liquid phase. Generally such particulate material will range in size from about 0.1 to 1500 microns, more preferably from about 0.1 to 900 microns. Most preferably, such material will range in size from about 5 to 200 microns.

The additional particulate material utilized herein can comprise one or more types of detergent composition components which in particulate form are substantially insoluble in the non-aqueous liquid phase of the composition. The types of particulate materials which can be utilized are described in detail as follows:

(a) Peroxygen Bleaching Agent With Optional Bleach Activators

The most preferred type of particulate material useful in the detergent compositions herein comprises particles of a peroxygen bleaching agent. Such peroxygen bleaching agents may be organic or inorganic in nature. Inorganic peroxygen bleaching agents are frequently utilized in combination with a bleach activator.

Useful organic peroxygen bleaching agents include percarboxylic acid bleaching agents and salts thereof. Suitable examples of this class of agents include magnesium monoperoxyphthalate hexahydrate, the magnesium salt of metachloro perbenzoic acid, 4-nonylamino-4-oxoperoxybutyric acid and diperoxydodecanedioic acid. Such bleaching agents

are disclosed in U.S. Patent 4,483,781, Hartman, Issued November 20, 1984; European Patent Application EP-A-133,354, Banks et al., Published February 20, 1985; and U.S. Patent 4,412,934, Chung et al., Issued November 1, 1983. Highly preferred bleaching agents also include 6-nonylamino-6-oxoperoxycaproic acid (NAPAA) as described in U.S. Patent 4,634,551, Issued January 6, 1987 to Burns et al.

Inorganic peroxygen bleaching agents may also be used in particulate form in the detergent compositions herein. Inorganic bleaching agents are in fact preferred. Such inorganic peroxygen compounds include alkali metal perborate and percarbonate materials, most preferably the percarbonates. For example, sodium perborate (e.g. mono- or tetra-hydrate) can be used. Suitable inorganic bleaching agents can also include sodium or potassium carbonate peroxyhydrate and equivalent "percarbonate" bleaches, sodium pyrophosphate peroxyhydrate, urea peroxyhydrate, and sodium peroxide. Persulfate bleach (e.g., OXONE, manufactured commercially by DuPont) can also be used. Frequently inorganic peroxygen bleaches will be coated with silicate, borate, sulfate or water-soluble surfactants. For example, coated percarbonate particles are available from various commercial sources such as FMC, Solvay Interlox, Tokai Denka and Degussa.

Inorganic peroxygen bleaching agents, e.g., the perborates, the percarbonates, etc., are preferably combined with bleach activators, which lead to the *in situ* production in aqueous solution (i.e., during use of the compositions herein for fabric laundering/bleaching) of the peroxy acid corresponding to the bleach activator. Various non-limiting examples of activators are disclosed in U.S. Patent 4,915,854, Issued April 10, 1990 to Mao et al.; and U.S. Patent 4,412,934 Issued November 1, 1983 to Chung et al. The nonanoyloxybenzene sulfonate (NOBS) and tetraacetyl ethylene diamine (TAED) activators are typical. Mixtures thereof can also be used. See also the hereinbefore referenced U.S. 4,634,551 for other typical bleaches and activators useful herein.

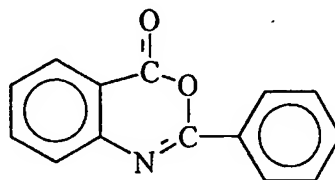
Other useful amido-derived bleach activators are those of the formulae:



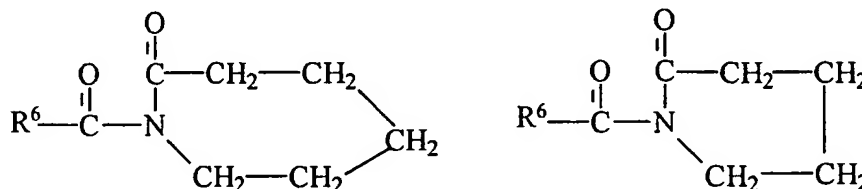
wherein R^1 is an alkyl group containing from about 6 to about 12 carbon atoms, R^2 is an alkylene containing from 1 to about 6 carbon atoms, R^5 is H or alkyl, aryl, or alkaryl containing from about 1 to about 10 carbon atoms, and L is any suitable leaving group. A leaving group is any group that is displaced from the bleach activator as a consequence of the nucleophilic attack on the bleach activator by the perhydrolysis anion. A preferred leaving group is phenol sulfonate.

Preferred examples of bleach activators of the above formulae include (6-octanamido-caproyl)oxybenzenesulfonate, (6-nonanamidocaproyl) oxybenzenesulfonate, (6-decanamidocaproyl)oxybenzenesulfonate and mixtures thereof as described in the hereinbefore referenced U.S. Patent 4,634,551. Such mixtures are characterized herein as (6-C₈-C₁₀ alkamido-caproyl)oxybenzenesulfonate.

Another class of useful bleach activators comprises the benzoxazin-type activators disclosed by Hodge et al. in U.S. Patent 4,966, 723, Issued October 30, 1990, incorporated herein by reference. A highly preferred activator of the benzoxazin-type is:



Still another class of useful bleach activators includes the acyl lactam activators, especially acyl caprolactams and acyl valerolactams of the formulae:



wherein R⁶ is H or an alkyl, aryl, alkoxyaryl, or alkaryl group containing from 1 to about 12 carbon atoms. Highly preferred lactam activators include benzoyl caprolactam, octanoyl caprolactam, 3,5,5-trimethylhexanoyl caprolactam, nonanoyl caprolactam, decanoyl caprolactam, undecenoyl caprolactam, benzoyl valerolactam, octanoyl valerolactam, decanoyl valerolactam, undecenoyl valerolactam, 3,5,5-trimethylhexanoyl valerolactam and mixtures thereof. See also U.S. Patent 4,545,784, Issued to Sanderson, October 8, 1985, incorporated herein by reference, which discloses acyl caprolactams, including benzoyl caprolactam, adsorbed into sodium perborate.

If peroxygen bleaching agents are used as all or part of the additional particulate material, they will generally comprise from about 1% to 30% by weight of the composition. More preferably, peroxygen bleaching agent will comprise from about 1% to 20% by weight of the composition. Most preferably, peroxygen bleaching agent will be present to the extent of from about 5% to 20% by weight of the composition. If utilized, bleach activators can comprise from about 0.5% to 20%, more preferably from about 3% to 10%, by weight of the composition. Frequently, activators are employed such that the molar ratio of bleaching agent to activator ranges from about 1:1 to 10:1, more preferably from about 1.5:1 to 5:1.

In addition, it has been found that bleach activators, when agglomerated with certain acids such as citric acid, are more chemically stable.

(b) Organic Builder Material

Another possible type of additional particulate material which can be suspended in the non-aqueous liquid detergent compositions herein comprises an organic detergent builder material which serves to counteract the effects of calcium, or other ion, water hardness encountered during laundering/bleaching use of the compositions herein. Examples of such materials include the alkali metal, citrates, succinates, malonates, fatty acids, carboxymethyl succinates, carboxylates, polycarboxylates and polyacetyl carboxylates. Specific examples include sodium, potassium and lithium salts of oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids and citric acid. Other examples of organic phosphonate type sequestering agents such as those which have been sold by Monsanto under the Dequest tradename and alkanehydroxy phosphonates. Citrate salts are highly preferred.

Other suitable organic builders include the higher molecular weight polymers and copolymers known to have builder properties. For example, such materials include appropriate polyacrylic acid, polymaleic acid, and polyacrylic/polymaleic acid copolymers and their salts, such as those sold by BASF under the Sokalan trademark which have molecular weight ranging from about 5,000 to 100,000.

Another suitable type of organic builder comprises the water-soluble salts of higher fatty acids, i.e., "soaps". These include alkali metal soaps such as the sodium, potassium, ammonium, and alkylammonium salts of higher fatty acids containing from about 8 to about 24 carbon atoms, and preferably from about 12 to about 18 carbon atoms. Soaps can be made by direct saponification of fats and oils or by the neutralization of free fatty acids. Particularly useful are the sodium and potassium salts of the mixtures of fatty acids derived from coconut oil and tallow, i.e., sodium or potassium tallow and coconut soap.

If utilized as all or part of the additional particulate material, insoluble organic detergent builders can generally comprise from about 2% to 20% by weight of the compositions herein. More preferably, such builder material can comprise from about 4% to 10% by weight of the composition.

(c) Inorganic Alkalinity Sources

Another possible type of additional particulate material which can be suspended in the non-aqueous liquid detergent compositions herein can comprise a material which serves to render aqueous washing solutions formed from such compositions generally alkaline in nature.

Such materials may or may not also act as detergent builders, i.e., as materials which counteract the adverse effect of water hardness on detergency performance.

Examples of suitable alkalinity sources include water-soluble alkali metal carbonates, bicarbonates, borates, silicates and metasilicates. Although not preferred for ecological reasons, water-soluble phosphate salts may also be utilized as alkalinity sources. These include alkali metal pyrophosphates, orthophosphates, polyphosphates and phosphonates. Of all of these alkalinity sources, alkali metal carbonates such as sodium carbonate are the most preferred.

The alkalinity source, if in the form of a hydratable salt, may also serve as a desiccant in the non-aqueous liquid detergent compositions herein. The presence of an alkalinity source which is also a desiccant may provide benefits in terms of chemically stabilizing those composition components such as the peroxygen bleaching agent which may be susceptible to deactivation by water.

If utilized as all or part of the additional particulate material component, the alkalinity source will generally comprise from about 1% to 25% by weight of the compositions herein. More preferably, the alkalinity source can comprise from about 2% to 15% by weight of the composition. Such materials, while water-soluble, will generally be insoluble in the non-aqueous detergent compositions herein. Thus such materials will generally be dispersed in the non-aqueous liquid phase in the form of discrete particles.

(d) Colored Speckles

The non-aqueous liquid detergent compositions herein also essentially contain from about 0.05% to 2%, more preferably 0.1% to 1%, of the composition of colored speckles. Such colored speckles themselves are combinations of a conventional dye or pigment material with a certain kind of carrier material that imparts specific characteristics to the speckles. For purposes of this invention, "colored" speckles are those which have a color that is visibly distinct from the color of the liquid detergent composition in which they are dispersed.

The colorant materials which can be used to form the colored speckles can comprise any of the conventional dyes and pigments known and approved for use in detergent products for use in the home. Such materials can include, for example, Ultramarine Blue dye, Acid 80 Blue dye, Red HP Liquitint, Blue Liquitint and the like.

Dye or pigment material can be combined with a specific type of carrier material to form the colored speckles for use in the detergent compositions herein. The carrier material is selected to impart to the speckles certain specific density and solubility characteristics. Materials which have been found to be suitable as carriers for the colored speckles include polyacrylates; polysaccharides such as starches, celluloses, gums and derivatives thereof; and polyethylene glycols. Especially preferred carrier material comprises polyethylene glycol having a molecular weight from about 4,000 to 20,000, more preferably from about 4,000 to 10,000.

The colored speckles can be produced by dispersing the dye or pigment material within the carrier material. This can be done, for example, by a) melting the carrier and dispersing the dye or pigment therein under mixing, b) mixing the dye/pigment powder and carrier powder together, or c) by dissolving the dye/pigment and the carrier in aqueous solution. The colorant/carrier mixture can then be formed into particles by flaking, spray drying, prilling, extruding or other conventional techniques. Generally the colored speckles will contain from about 0.1% to 5% by weight of the speckles of the colorant (dye or pigment) material.

The colored speckles produced in this manner will generally range in size from about 400 to 1,500 microns, more preferably from about 400 to 1,200 microns. Speckles made from the carrier materials specified will have a density less than about 1.4 g/cc, preferably from about 1.0 to 1.4 g/cc. Such speckles will also be substantially insoluble in the non-aqueous liquid phase of the liquid detergent compositions herein. Thus, the colored speckles can be stably suspended in the non-aqueous matrix of the liquid detergent compositions of this invention without dissolving therein. Such speckles, however, rapidly dissolve in the aqueous wash liquors prepared from the liquid detergent compositions herein.

OTHER OPTIONAL COMPOSITION COMPONENTS

In addition to the composition liquid and solid phase components as hereinbefore described, the detergent compositions herein can, and preferably will, contain various other optional components. Such optional components may be in either liquid or solid form. The optional components may either dissolve in the liquid phase or may be dispersed within the liquid phase in the form of fine particles or droplets. Some of the other materials which may optionally be utilized in the compositions herein are described in greater detail as follows:

(a) Optional Inorganic Detergent Builders

The detergent compositions herein may also optionally contain one or more types of inorganic detergent builders beyond those listed hereinbefore that also function as alkalinity sources. Such optional inorganic builders can include, for example, aluminosilicates such as zeolites. Aluminosilicate zeolites, and their use as detergent builders are more fully discussed in Corkill et al., U.S. Patent No. 4,605,509; Issued August 12, 1986, the disclosure of which is incorporated herein by reference. Also crystalline layered silicates, such as those discussed in this '509 U.S. patent, are also suitable for use in the detergent compositions herein. If utilized, optional inorganic detergent builders can comprise from about 2% to 15% by weight of the compositions herein.

(b) Optional Enzymes

The detergent compositions herein may also optionally contain one or more types of detergent enzymes. Such enzymes can include proteases, amylases, cellulases and lipases. Such materials are known in the art and are commercially available. They may be incorporated into the non-aqueous liquid detergent compositions herein in the form of suspensions, "marumes" or "prills". Another suitable type of enzyme comprises those in the form of slurries of enzymes in nonionic surfactants, e.g., the enzymes marketed by Novo Nordisk under the tradename "SL" or the microencapsulated enzymes marketed by Novo Nordisk under the tradename "LDP."

Enzymes added to the compositions herein in the form of conventional enzyme prills are especially preferred for use herein. Such prills will generally range in size from about 100 to 1,000 microns, more preferably from about 200 to 800 microns and will be suspended throughout the non-aqueous liquid phase of the composition. Prills in the compositions of the present invention have been found, in comparison with other enzyme forms, to exhibit especially desirable enzyme stability in terms of retention of enzymatic activity over time. Thus, compositions which utilize enzyme prills need not contain conventional enzyme stabilizing such as must frequently be used when enzymes are incorporated into aqueous liquid detergents.

If employed, enzymes will normally be incorporated into the non-aqueous liquid compositions herein at levels sufficient to provide up to about 10 mg by weight, more typically

from about 0.01 mg to about 5 mg, of active enzyme per gram of the composition. Stated otherwise, the non-aqueous liquid detergent compositions herein will typically comprise from about 0.001% to 5%, preferably from about 0.01% to 1% by weight, of a commercial enzyme preparation. Protease enzymes, for example, are usually present in such commercial preparations at levels sufficient to provide from 0.005 to 0.1 Anson units (AU) of activity per gram of composition.

(c) Optional Chelating Agents

The detergent compositions herein may also optionally contain a chelating agent which serves to chelate metal ions, e.g., iron and/or manganese, within the non-aqueous detergent compositions herein. Such chelating agents thus serve to form complexes with metal impurities in the composition which would otherwise tend to deactivate composition components such as the peroxygen bleaching agent. Useful chelating agents can include amino carboxylates, phosphonates, amino phosphonates, polyfunctionally-substituted aromatic chelating agents and mixtures thereof.

Amino carboxylates useful as optional chelating agents include ethylenediaminetetraacetates, N-hydroxyethyl-ethylenediaminetriacetates, nitrilotriacetates, ethylene-diamine tetrapropionates, triethylenetetraaminehexacetates, diethylenetriaminepentaacetates, ethylenediaminedisuccinates and ethanol diglycines. The alkali metal salts of these materials are preferred.

Amino phosphonates are also suitable for use as chelating agents in the compositions of this invention when at least low levels of total phosphorus are permitted in detergent compositions, and include ethylenediaminetetrakis (methylene-phosphonates) as DEQUEST. Preferably, these amino phosphonates do not contain alkyl or alkenyl groups with more than about 6 carbon atoms.

Preferred chelating agents include hydroxy-ethyldiphosphonic acid (HEDP), diethylene triamine penta acetic acid (DTPA), ethylenediamine disuccinic acid (EDDS) and dipicolinic acid (DPA) and salts thereof. The chelating agent may, of course, also act as a detergent builder during use of the compositions herein for fabric laundering/bleaching. The chelating agent, if employed, can comprise from about 0.1% to 4% by weight of the compositions herein. More preferably, the chelating agent will comprise from about 0.2% to 2% by weight of the detergent compositions herein.

(d) Optional Thickening, Viscosity Control and/or Dispersing Agents

The detergent compositions herein may also optionally contain a polymeric material which serves to enhance the ability of the composition to maintain its solid particulate

components in suspension. Such materials may thus act as thickeners, viscosity control agents and/or dispersing agents. Such materials are frequently polymeric polycarboxylates but can include other polymeric materials such as polyvinylpyrrolidone (PVP) or polyamide resins.

Polymeric polycarboxylate materials can be prepared by polymerizing or copolymerizing suitable unsaturated monomers, preferably in their acid form. Unsaturated monomeric acids that can be polymerized to form suitable polymeric polycarboxylates include acrylic acid, maleic acid (or maleic anhydride), fumaric acid, itaconic acid, aconitic acid, mesaconic acid, citraconic acid and methylenemalononic acid. The presence in the polymeric polycarboxylates herein of monomeric segments, containing no carboxylate radicals such as vinylmethyl ether, styrene, ethylene, etc. is suitable provided that such segments do not constitute more than about 40% by weight of the polymer.

Particularly suitable polymeric polycarboxylates can be derived from acrylic acid. Such acrylic acid-based polymers which are useful herein are the water-soluble salts of polymerized acrylic acid. The average molecular weight of such polymers in the acid form preferably ranges from about 2,000 to 100,000, more preferably from about 2,000 to 10,000, even more preferably from about 4,000 to 7,000, and most preferably from about 4,000 to 5,000. Water-soluble salts of such acrylic acid polymers can include, for example, the alkali metal, salts. Soluble polymers of this type are known materials. Use of polyacrylates of this type in detergent compositions has been disclosed, for example, Diehl, U.S. Patent 3,308,067, issued March 7, 1967. Such materials may also perform a builder function.

Other suitable polymeric materials suitable for use as thickening, viscosity control and/or dispersing agents include polymers of: castor oil derivatives; polyurethane derivatives, and polyethylene glycol.

If utilized, the optional thickening, viscosity control and/or dispersing agents should be present in the compositions herein to the extent of from about 0.1% to 4% by weight. More preferably, such materials can comprise from about 0.1% to 2% by weight of the detergents compositions herein.

(e) Optional Clay Soil Removal/Anti-redeposition Agents

The compositions of the present invention can also optionally contain water-soluble ethoxylated amines having clay soil removal and anti-redeposition properties. If used, soil materials can contain from about 0.01% to about 5% by weight of the compositions herein.

The most preferred soil release and anti-redeposition agent is ethoxylated tetraethylenepentamine. Exemplary ethoxylated amines are further described in U.S. Patent 4,597,898, VanderMeer, issued July 1, 1986. Another group of preferred clay soil removal-anti-redeposition agents are the cationic compounds disclosed in European Patent Application 111,965, Oh and Gosselink, published June 27, 1984. Other clay soil removal/anti-redeposition agents which can be used include the ethoxylated amine polymers disclosed in European Patent Application 111,984, Gosselink, published June 27, 1984; the zwitterionic polymers disclosed in European Patent Application 112,592, Gosselink, published July 4, 1984; and the amine oxides disclosed in U.S. Patent 4,548,744, Connor, issued October 22, 1985. Other clay soil removal and/or anti-redeposition agents known in the art can also be utilized in the compositions herein. Another type of preferred anti-redeposition agent includes the carboxy methyl cellulose (CMC) materials. These materials are well known in the art.

(f) Optional Liquid Bleach Activators

The detergent compositions herein may also optionally contain bleach activators which are liquid in form at room temperature and which can be added as liquids to the non-aqueous liquid phase of the detergent compositions herein. One such liquid bleach activator is acetyl triethyl citrate (ATC). Other examples include glycerol triacetate and nonanoyl valerolactam. Liquid bleach activators can be dissolved in the non-aqueous liquid phase of the compositions herein.

(g) Optional Brighteners, Suds Suppressors, Dyes and/or Perfumes

The detergent compositions herein may also optionally contain conventional brighteners, suds suppressors, bleach catalysts, dyes and/or perfume materials. Such brighteners, suds suppressors, silicone oils, bleach catalysts, dyes and perfumes must, of course, be compatible and non-reactive with the other composition components in a non-aqueous environment. If present, brighteners suds suppressors, dyes and/or perfumes will typically comprise from about 0.0001% to 2% by weight of the compositions herein. Suitable bleach catalysts include the manganese based complexes disclosed in US 5,246,621, US 5,244,594, US 5,114,606 and US 5,114,611.

(h) Structure Elasticizing Agents

The non-aqueous liquid detergent compositions herein can also contain from about 0.1% to 5%, preferably from about 0.1% to 2% by weight of a finely divided, solid particulate material which can include silica, e.g., fumed silica, titanium dioxide, insoluble carbonates, finely divided carbon, SD-3 bentone, clays, or combinations of these materials. Clays are well known to those skilled in the art and are commercially available from companies such as Rheox. Fine particulate material of this type functions as a structure elasticizing agent in the products of this invention. Such material has an average particle size ranging from about 7 to 40 nanometers, more preferably from about 7 to 15 nanometers. Such material also has a specific surface area which ranges from about 40 to 400m²/g.

The finely divided elasticizing agent material can improve the shipping stability of the non-aqueous liquid detergent products herein by increasing the elasticity of the surfactant-structured liquid phase without increasing product viscosity. This permits such products to withstand high frequency vibration which may be encountered during shipping without undergoing undesirable structure breakdown which could lead to sedimentation in the product.

In the case of titanium dioxide, the use of this material also imparts whiteness to the suspension of particulate material within the detergent compositions herein. This effect improves the overall appearance of the product.

COMPOSITION FORM

As indicated, the non-aqueous liquid detergent compositions herein are in the form of bleaching agent and/or other materials in particulate form as a solid phase suspended in and dispersed throughout a surfactant-containing, preferably structured non-aqueous liquid phase. Generally, the structured non-aqueous liquid phase will comprise from about 45% to 95%, more preferably from about 50% to 90%, by weight of the composition with the dispersed additional solid materials comprising from about 5% to 55%, more preferably from about 10% to 50%, by weight of the composition.

The particulate-containing liquid detergent compositions of this invention are substantially non-aqueous (or anhydrous) in character. While very small amounts of water may be incorporated into such compositions as an impurity in the essential or optional components, the amount of water should in no event exceed about 5% by weight of the compositions herein. More preferably, water content of the non-aqueous detergent compositions herein will comprise less than about 1% by weight.

The particulate-containing non-aqueous liquid detergent compositions herein will be relatively viscous and phase stable under conditions of commercial marketing and use of such compositions. Frequently the viscosity of the compositions herein will range from about 300 to

5,000 cps, more preferably from about 500 to 3,000 cps. For purposes of this invention, viscosity is measured with a Carrimed CSL2 Rheometer at a shear rate of 20 s^{-1} .

COMPOSITION PREPARATION AND USE

The non-aqueous liquid detergent compositions herein can be prepared by first forming the surfactant-containing, preferably structured non-aqueous liquid phase and by thereafter adding to this structured phase the gasified particles of this invention and additional particulate components in any convenient order and by mixing, e.g., agitating, the resulting component combination to form the phase stable compositions herein. It is generally preferred, however, to add the gasified particles near the end of the detergent processing to avoid unnecessary milling of the gasified particles. It is understood that as the gasified particles are broken or crushed in a milling or other size reduction processing step, a certain amount of the gas is released. This can reduce the efficiency of the gasified particles.

In a typical process for preparing the compositions of this invention, essential and certain preferred optional components will be combined in a particular order and under certain conditions. In a first step of a preferred preparation process, the anionic surfactant-containing powder used to form the structured, surfactant-containing liquid phase is prepared. This pre-preparation step involves the formation of an aqueous slurry containing from about 30% to 60% of one or more alkali metal salts of linear C_{10-16} alkyl benzene sulfonic acid and from about 2% to 30% of one or more diluent non-surfactant salts. In a subsequent step, this slurry is dried to the extent necessary to form a solid material containing less than about 4% by weight of residual water.

After preparation of this solid anionic surfactant-containing material, this material can be combined with one or more of the non-aqueous organic diluents to form a structured, surfactant-containing liquid phase of the detergent compositions herein. This is done by reducing the anionic surfactant-containing material formed in the previously described pre-preparation step to powdered form and by combining such powdered material with an agitated liquid medium comprising one or more of the non-aqueous organic diluents, either surfactant or non-surfactant or both, as hereinbefore described. This combination is carried out under agitation conditions which are sufficient to form a thoroughly mixed dispersion of particles of the insoluble fraction of the co-dried LAS/salt material throughout a non-aqueous organic liquid diluent.

In a subsequent processing step, the non-aqueous liquid dispersion so prepared can then be subjected to milling or high shear agitation under conditions which are sufficient to provide a structured, surfactant-containing liquid phase of the detergent compositions herein. Such milling or high shear agitation conditions will generally include maintenance of a temperature between about 10°C and 90°C , preferably between about 20°C and 60°C ; and a processing time

that is sufficient to form a network of aggregated small particles of the insoluble fraction of the anionic surfactant-containing powdered material. Suitable equipment for this purpose includes: stirred ball mills, co-ball mills (Fryma), colloid mills, high pressure homogenizers, high shear mixers, and the like. The colloid mill and high shear mixers are preferred for their high throughput and low capital and maintenance costs. The small particles produced in such equipment will generally range in size from about 0.4 to 2 microns. Milling and high shear agitation of the liquid/solids combination will generally provide an increase in the yield value of the structured liquid phase to within the range of from about 1 Pa to 8 Pa, more preferably from about 2 Pa to 6 Pa.

After formation of the dispersion of LAS/salt co-dried material in the non-aqueous liquid, either before or after such dispersion is milled or agitated to increase its yield value, the gasified particles of this invention and the additional particulate material to be used in the detergent compositions herein can be added. Such components which can be added under high shear agitation include the gasified particles; a silica or titanium dioxide elasticizing agent; particles of substantially all of an organic builder, e.g., citrate and/or fatty acid, and/or an alkalinity source, e.g., sodium carbonate, can be added while continuing to maintain this admixture of composition components under shear agitation. Agitation of the mixture is continued, and if necessary, can be increased at this point to form a uniform dispersion of insoluble solid phase particulates within the liquid phase.

After some or all of the foregoing solid materials have been added to this agitated mixture, the particles of the highly preferred peroxygen bleaching agent can be added to the composition, again while the mixture is maintained under shear agitation. By adding the peroxygen bleaching agent material last, or after all or most of the other components, and especially after alkalinity source particles, have been added, desirable stability benefits for the peroxygen bleach can be realized. If enzyme prills are incorporated, they are preferably added to the non-aqueous liquid matrix last.

As a final process step, after addition of all of the particulate material, agitation of the mixture is continued for a period of time sufficient to form compositions having the requisite viscosity, yield value and phase stability characteristics. Frequently this will involve agitation for a period of from about 1 to 30 minutes.

In adding solid components to non-aqueous liquids in accordance with the foregoing procedure, it is advantageous to maintain the free, unbound moisture content of these solid materials below certain limits. Free moisture in such solid materials is frequently present at levels of 0.8% or greater. By reducing free moisture content, e.g., by fluid bed drying, of solid particulate materials to a free moisture level of 0.5% or lower prior to their incorporation into

the detergent composition matrix, significant stability advantages for the resulting composition can be realized.

The compositions of this invention, prepared as hereinbefore described, can be used to form aqueous washing solutions for use in the laundering and bleaching of fabrics. Generally, an effective amount of such compositions is added to water, preferably in a conventional fabric laundering automatic washing machine, to form such aqueous laundering/bleaching solutions. The aqueous washing/bleaching solution so formed is then contacted, preferably under agitation, with the fabrics to be laundered and bleached therewith.

An effective amount of the liquid detergent compositions herein added to water to form aqueous laundering/bleaching solutions can comprise amounts sufficient to form from about 500 to 7,000 ppm of composition in aqueous solution. More preferably, from about 800 to 3,000 ppm of the detergent compositions herein will be provided in aqueous washing/bleaching solution.

The following examples illustrate the preparation and performance advantages of the speckle-containing non-aqueous liquid detergent compositions of the instant invention. Such examples, however, are not necessarily meant to limit or otherwise define the scope of the invention herein.

EXAMPLE I

Preparation of Gasified Particles of PEG and N₂

Gasified Particles for use in the non-aqueous liquid detergent composition hereinafter described are prepared from nitrogen gas and polyethylene glycol with a molecular weight of about 4,000, i.e., PEG-4000. To prepare these particles, the PEG-4000 is melted in a pressurizable vessel while agitation is supplied in the form of a mixing paddle attached to a shaft. Nitrogen gas is added to the pressure vessel from ports in the top of the vessel and through ports in the shaft that holds the mixing paddle. The vessel is pressurized to about 600 psig with the nitrogen gas. When the PEG-4000 is sufficiently melted and mixed with pressurized gas, the molten PEG-4000 and nitrogen mixture is transferred to a cooling vessel. The mixture is cooled until it solidifies, and then the solidified material is removed from the cooling vessel by breaking it into small pieces. The small pieces are then milled to form particles having a size ranging from about 0.1 to about 1000 microns.

EXAMPLE II**Preparation of Gasified Particles of Sucrose and CO₂**

The process of Example I is repeated, wherein the PEG-4000 is replaced with sucrose and the nitrogen gas is replaced with carbon dioxide.

EXAMPLE III**Preparation of LAS Powder for Use as a Structurant**

Sodium C₁₂ linear alkyl benzene sulfonate (NaLAS) is processed into a powder containing two phases. One of these phases is soluble in the non-aqueous liquid detergent compositions herein and the other phase is insoluble. It is the insoluble fraction which serves to add structure and particle suspending capability to the non-aqueous phase of the compositions herein.

NaLAS powder is produced by taking a slurry of NaLAS in water (approximately 40-50% active) combined with dissolved sodium sulfate (3-15%) and hydrotrope, sodium sulfosuccinate (1-3%). The hydrotrope and sulfate are used to improve the characteristics of the dry powder. A drum dryer is used to dry the slurry into a flake. When the NaLAS is dried with the sodium sulfate, two distinct phases are created within the flake. The insoluble phase creates a network structure of aggregate small particles (0.4-2 μ m) which allows the finished non-aqueous detergent product to stably suspend solids.

The NaLAS powder prepared according to this example has the following makeup shown in Table III.

TABLE III**LAS Powder**

<u>Component</u>	<u>Wt. %</u>
NaLAS	85%
Sulfate	11%
Sulfosuccinate	2%
Water	2.5%
Unreacted, etc.	balance to 100%
% insoluble LAS	17%
# of phase (via X-ray diffraction)	2

EXAMPLE IV**Preparation of Non-Aqueous Liquid Detergent Composition**

- 1) Butoxy-propoxy-propanol (BPP) and a C₁₁₋₁₅EO(5) ethoxylated alcohol nonionic surfactant (Neodol 1-5) are mixed for a short time (1-2 minutes) using a pitched blade turbine impeller in a mix tank into a single phase.
- 2) NaLAS powder as prepared in Example III is added to the BPP/Neodol solution in the mix tank to partially dissolve the NaLAS. Mix time is approximately one hour. The tank is blanketed with nitrogen to prevent moisture pickup from the air. The soluble phase of NaLAS powder dissolves, while the insoluble NaLAS aggregates and forms a network structure within the BPP/Neodol solution.
- 3) Liquid base (LAS/BPP/NI) is pumped out into drums. Molecular sieves (type 3A, 4-8 mesh) are added to each drum at 10% of the net weight of the liquid base. The molecular sieves are mixed into the liquid base using both single blade turbine mixers and drum rolling techniques. The mixing is done under nitrogen blanket to prevent moisture pickup from the air. Total mix time is 2 hours, after which 0.1-0.4% of the moisture in the liquid base is removed.
- 4) Molecular sieves are removed by passing the liquid base through a 20-30 mesh screen. Liquid base is returned to the mix tank.
- 5) Additional solid ingredients are prepared for addition to the composition. Such solid ingredients include the following:

Sodium carbonate (particle size 10-40 microns)

Sodium citrate dihydrate

Maleic-acrylic copolymer (BASF's Sokalan CP5; moisture content 4.1-5.0%)

Brightener

Titanium dioxide particles (1-5 microns)

Trisodium ethylene diamine disuccinate (EDDS)

These solid materials, which are all millable, are added to the mix tank through a 20-30 mesh screen and mixed with the liquid base until smooth. This approximately 1 hour after addition of the last powder. The tank is blanketed with nitrogen after addition of the powders. No particular order of addition for these powders is critical.

- 6) The batch is pumped once through a Fryma colloid mill, which is a simple rotor-stator configuration in which a high-speed rotor spins inside a stator which creates a zone of high shear. This serves to disperse the insoluble NaLAS aggregates and partially reduce the particle size of all of the solids. This leads to an increase in yield value (i.e. structure). The batch is then recharged to the mix tank.
- 7) Still additional solid materials which should not be milled or subjected to high shear agitation are then prepared. These include the following:

Gasified Particles from Example I

Sodium 6-(C₈₋₁₀ alkamidocaproyl) oxybenzene sulfonate bleach activator

Sodium perborate (20-40 microns)

Cellulase and amylase enzyme prills (100-1000 microns)

Thickener

Ethoxylated hexamethylenediamine quat

These non-millable solid materials are then added to the mix tank followed by liquid ingredients (perfume and silicone-based suds suppressor). The batch is then mixed for one hour (under nitrogen blanket). The resulting composition has the formula set forth in Table IV.

TABLE IV

Non-Aqueous Liquid Detergent Composition with Bleach

<u>Component</u>	<u>Wt % Active</u>
LAS	16
C ₁₂₋₁₄ E ₀₌₅ alcohol ethoxylate	22
BPP	19
Sodium citrate dihydrate	3
Bleach activator	5.9
Sodium carbonate	9
Maleic-acrylic copolymer	3
Gasified Particles	0.4
EDDS	1
Cellulase Prills	0.12
Amylase Prills	0.4
Ethoxylated diamine quat	1.25

Sodium Perborate	15
Thickener	0.4
Suds suppressor	0.04
Perfume	0.48
Titanium dioxide	0.5
Brightener	0.2
Sulfate	<u>2.31</u>
	100.00%

The resulting Table IV composition is a stable, anhydrous heavy-duty liquid laundry detergent which provides excellent stain and soil removal performance when used in normal fabric laundering operations. It has the desired gasified particles of this invention suspended throughout a generally white opaque liquid composition.

WHAT IS CLAIMED IS:

1. A non-aqueous liquid detergent composition which is in the form of a suspension of solid, substantially insoluble particulate material dispersed throughout a surfactant-containing non-aqueous liquid phase, which composition comprises:
 - A) from about 49% to 99.95% by weight of the composition of a surfactant-containing non-aqueous liquid phase; and
 - B) from about 0.05% to about 5%, preferably from about 0.1% to about 3%, and most preferably from about 0.2% to about 2%, by weight of the composition of gasified particles that are:
 - i) substantially insoluble in the non-aqueous liquid phase;
 - ii) solid at about 25°C; and
 - iii) highly water soluble.
2. A composition according to Claim 1, wherein the surfactant-containing liquid phase is formed by combining:
 - i) from about 1% to about 80% by weight of the liquid phase of one or more non-aqueous organic diluents; and
 - ii) from about 20% to 99% by weight of the liquid phase of a surfactant selected from anionic, nonionic and cationic surfactants and combinations thereof.
3. A composition according to Claim 1, wherein the gasified particles comprise a core material that encapsulates a pressurized gas.
4. A composition according to Claim 3, wherein the pressurized gas is selected from the group consisting of carbon dioxide, nitrogen, oxygen, helium, hydrogen, air, argon, neon, chlorine and mixtures thereof.
5. A composition according to Claim 3, wherein the core material is selected from the group consisting of sucrose, lactose, glucose, fructose, galactose, maltose, polyethylene glycol, polyvinyl alcohol, fatty acids, and mixtures thereof.
6. A composition according to Claim 3, wherein the pressurized gas is present in the gasified particles at a pressure of from about 50 psig to about 1,000 psig, preferably from about 300 psig to about 1,000 psig, most preferably from about 600 psig to about 1,000 psig.

7. A composition according to Claim 3, wherein the gasified particles additionally comprise a dye or pigment, preferably selected from the group consisting of ultramarine blue, indigo carmine, FD&C blue 1, D&C yellow 5, D&C yellow 6, D&C red 21, D&C red 27, D&C orange 5, bromo acid dyes, sodium fluorescein, liquitint bright blue, liquitint bright yellow, duasyn blue and mixtures thereof.
8. A composition according to Claim 3, wherein the gasified particles additionally comprise a water soluble or dispersable coating that preferably comprises materials selected from the group consisting of sucrose, lactose, glucose, fructose, galactose, maltose, polyethylene glycol, polyvinyl alcohol, fatty acids, and mixtures thereof.
9. A composition according to Claim 1, wherein the composition further comprises from about 1% to 50% by weight of the composition of additional particulate material which ranges in size from about 0.1 to 1500 microns, which is substantially insoluble in said liquid phase and which is selected from peroxygen bleaching agents, bleach activators, organic detergent builders and inorganic alkalinity sources and combinations thereof.
10. A composition according to Claim 1, wherein the gasified particles range in particle size between about 0.1 and about 1,500 microns, preferably between about 1 and about 1,000 microns, and most preferably between about 10 and about 400 microns.
11. A composition according to Claim 1, wherein the gasified particles range in density between about 0.6 and 1.4 g/cc, more preferably between about 0.8 and 1.3 g/cc, and most preferably between about 1.0 and 1.3 g/cc.
12. A composition according to Claim 8, wherein the core material comprises polyethylene glycol having a molecular weight between about 2,000 and 20,000.
13. A composition according to Claim 1, wherein the surfactant-containing non-aqueous liquid phase has a density of from about 0.6 to 1.4 g/cc.
14. A composition according to Claim 3, wherein the gasified particles comprise between about 0.5 and 15 milliliters of pressurized gas per gram of core material.
15. A composition according to Claim 3, wherein the pressurized gas comprises a perfume.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/IB 99/00996

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D17/00 C11D3/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 32 24 970 A (STAEDTLER FA J S) 5 January 1984 (1984-01-05) claims 1-26 ---	1
A	EP 0 349 314 A (UNILEVER PLC ;UNILEVER NV (NL)) 3 January 1990 (1990-01-03) claims 1-15 ---	1
A	DE 39 03 598 A (COLGATE PALMOLIVE CO) 17 August 1989 (1989-08-17) claims 1-15 ---	1
A	US 4 592 855 A (GIOFFRE ANTHONY J ET AL) 3 June 1986 (1986-06-03) claims 1-16 ---	1
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents :

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"O" document referring to an oral disclosure, use, exhibition or other means

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Date of the actual completion of the international search

4 August 1999

Date of mailing of the international search report

19/08/1999

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INTERNATIONAL SEARCH REPORT

Int'l. Application No
PCT/IB 99/00996

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

Int lional Application No

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